GENERAL ATOMIC
Division of General Dynamics Corporation

John Jay Hopkins Laboratory
for Pure and Applied Science

GACD-4457
Copy No. 3

INVESTIGATIONS OF CARBIDES AS CATHODES FOR THERMIONIC SPACE REACTORS

Informal Monthly Report for the Period
June 1, 1963 through June 30, 1963

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Project No. 306
National Aeronautics and Space Administration
Contract No. NAS 3-2532

August 5, 1963

SUMMARY

The work carried out under Contract NAS 3-2532 during June, 1963 is summarized as follows:

1. Long-term Tests of the Vaporization and Emission of UC-ZrC and W-Clad UC in Cesium Vapor.

1.1 Rate of Vaporization of UC-ZrC in Cesium Vapor.

Two cells, one containing a 30 UC - 70 ZrC sample (B_1-9a) and the other containing a 90 UC - 10 ZrC sample (B_1-10) are in operation.

1.2 Emission Stability of UC-ZrC and W-Clad UC and UC-ZrC in Cesium Vapor.

The control cell containing a solid cast W emitter has been assembled and test of this cell is being pursued. Assembling of the four emission-life test cells is being continued.

2. Studies of the High-Temperature Properties of UC-ZrC.

2.1 Effect of Porosity on the Vaporization and Electron Emission Properties of UC-ZrC in Vacuum.

Due to the tieup of the Langmuir vaporization cell for the study of new cathode materials, no measurement was made during this reporting period.

2.2 Redeposition Studies.

The condensates from the physical redeposition runs (described in the 3rd quarterly report) of UC (sample B_2-1), 30 UC - 70 ZrC (sample B_1-9a) and 90 UC - 10 ZrC (sample B_1-10) cathodes versus Ni anode have been analyzed for their uranium and zirconium contents. Other physical redeposition studies of UC cathode versus Ni anode are in progress.

2.3 Thermionic Emission Microscopy.

The poisoning and regeneration of the electron emission of another hyperstoichiometric UC sample have been studied. The similarity between the emission pattern and the microstructures of the sample has been demonstrated.

2.4 High-Temperature Mechanical Properties.

The UC-ZrC samples needed for the tests have been prepared. The modified bearing for guiding the loading rod is under test.

3. Irradiation Studies.

3.1 Unclad Carbide Capsule.

Hot cell examinations of the samples in the unclad carbide capsule is underway.

3.2 Clad Capsule.

The clad capsule is being irradiated in the General Electric Test Reactor.

4. Studies of New Cathode Materials.

4.1 Vacuum Emission and Vaporization.

The vacuum emission of a 30 UC - 70 NbC sample (D_1-3) has been measured. Study of the vacuum rate of vaporization of a 30 UC - 70 TaC sample (D_1-4) is in progress.

4.2 Diffusion.

A 24 hour diffusion run (D_2-5) of Ir, Re and W-26Re versus hypo-stoichiometric 30 UC - 70 ZrC has been made at 1800°C.

4.3 Diffusion-Emission.

Diffusion-emission studies of vapor-deposited W clad UC (4.70 wt-% C) and 30 UC - 70 ZrC samples (D_3-2 and D_3-3) at 1800°C are in progress.

5. Fabrication Development.

Since the fabrication of the samples needed for the irradiation capsules and various measurements has essentially been completed, no work in fabrication development is planned for the rest of this contractual period.

I. EXPERIMENTAL PROGRAM.

This monthly progress report covers the work accomplished during the period June 1 to June 30, 1963 under Contract NAS 3-2532. The purpose of this contract is to continue the efforts made under Contract NAS 5-1253[1] and NAS 3-2310[2] for establishing the feasibility of the UC-ZrC and the W-clad UC and UC-ZrC thermionic cathode system and to develop new cathode materials. The subjects to be studied are (i) long-term (≥ 1000 hr) tests of the vaporization and emission stability of the UC-ZrC and the W-clad UC systems in the presence of cesium vapor; (ii) studies of the controlling factors of the vaporization, emission and mechanical properties of the UC-ZrC systems; (iii) studies of the irradiation properties of the UC-ZrC and the W-clad UC systems; (iv) studies of the vaporization, emission and diffusion properties of new cathode materials; and (v) development of fabrication techniques for controlling the structures and compositions of samples used in these studies.

1. Long-term Tests.

1.1 Rate of Vaporization of UC-ZrC in Cesium Environment.

The cell containing the 30 UC - 70 ZrC sample (B_1-9a) has been operated satisfactorily under the following conditions for a period of 200 hours at the end of this reporting period: carbide temperature, 2113°K Ni anode temperature, 983°K; spacing between carbide sample and Ni anode, 20 mils; cesium pressure, 1.8 torr. The operation of the second cell containing the 90 UC - 10 ZrC sample (B_1-10) has just been started under the following conditions: carbide temperature, 2075°K; Ni anode temperature, 983°K; spacing between carbide sample and Ni anode, 20 mils; cesium pressure, 1.8 torr. It is planned to continue these tests for periods of 1000 hours.

1.2 Emission Stability of UC-ZrC and W-Clad UC and UC-ZrC in Cesium Vapor.

The emission-life test control cell (MLP-C) started operation on June 25, 1963. This cell contains a solid case W emitter diffusion-bonded directly to the Ta holder of the emitter assembly. The purpose of this cell was to evaluate the improvements made in the electron gun filament design and the W-clad Ta emitter holder. In addition, the basic cell operation

characteristics could be optimized and the conditions under which an electrical output of ~5 watts/cm^2 determined.

Preliminary studies of the cell revealed that more control of the temperatures of the cesium reservoir and the collector was necessary to establish the conditions for the optimum output of the cell at given emitter temperatures. Two temperature control systems were therefore set up for this purpose. Studies of the cell output as a function of the cell operating conditions are being pursued. The data obtained will be analyzed and reported in the July, 1963 report.

2. Studies of the High-Temperature Properties of UC-ZrC.

2.1 Effect of Porosity on the Vaporization and Electon Emission Properties of UC-ZrC in Vacuum.

No work has been carried out on this section due to the tie up of the Langmuir vaporization cell for the measurement of the vacuum vaporization rate of new cathode materials. Three samples are ready for the study of their vacuum vaporization rate and electron emission properties. These are (i) 30 UC - 70 ZrC, ~95% dense; (ii) 30 UC - 70 ZrC, ~85% dense; (iii) 90 UC - 10 ZrC, ~85% dense. It is expected that the study will be resumed in July, 1963.

2.2 Redeposition Studies.

The condensates collected from the physical redeposition runs[3] of UC (sample B_2-1), 90 UC - 10 ZrC (sample B_1-10) and 30 UC - 70 ZrC (sample B_1-9a) versus Ni anode have been analyzed for their U and Zr contents. The results are summarized in Table I, together with other pertinent data of these runs.

It is interesting to note that for cases where the presence of anode did not affect the rate of vaporization, the amount of U and Zr collected by the anode can account for about 95% of the vaporization loss, indicating a very high capture of the vaporized materials by the anode. On the other hand, for cases where the presence of anode reduced the rate of vaporization, the fraction of vaporized materials captured by the anode is considerably less, indicating back scattering of the vaporized atoms by the anode surface and the loss of part of the back scattered atoms from the interelectrode

space to the cold wall of the cell. No anode effect was observed for the 30 UC - 70 ZrC sample even when the anode was at 983°K, although for 90 UC - 10 ZrC and for UC, the vaporization losses were reduced by about a factor of 2 under such conditions. As hypothesized in the third quarterly report,(3) this is probably due to the presence of more Zr atoms in the vaporized materials from the 30 UC - 70 ZrC sample than that from the 90 UC - 10 ZrC sample, which, due to its low vapor pressure, will tend to condense more easily than U. Presumably, the presence of Zr on the anode surface helps the condensation of U and C and thus nullified the anode effect.

Studies of the physical redeposition of UC versus Ni anode are being carried out at anode temperature higher than 983°K. In addition, the compatibility of CsF with Cu and Ni is being checked at 500°C in order to find out whether a Cu or a Ni reservoir could be used for the CsF additive during chemical redeposition studies.

2.3 Thermionic Emission Microscopy.

Studies of the poisoning and regeneration of the electron emission of UC have been made with another hyperstoichiometric sample in the temperature range 1650 - 2000°K. The results obtained confirm that described in the third quarterly report. The emission patterns remained to be patchy even at 2000°K. Poison of the emission occurred upon exposure to ambient vacuum of about 10^{-2} torr but recovered within a few hours upon heating to 1770°K in a vacuum of 10^{-6} torr. Good correlation was obtained between the emission pattern and the microphotographs obtained. A cast 90 UC - 10 ZrC sample is ready for similar studies.

2.4 High-Temperature Mechanical Properties.

The modified bearing for guiding the loading rod has been mounted to the top of the furnace and is now under testing. The UC-ZrC samples needed for the studies have been fabricated. These involve twelve 10 UC - 90 ZrC samples, nine 30 UC - 70 ZrC samples and eight 80 UC - 20 ZrC samples.

3. Irradiation Studies.

3.1 Unclad Carbide Capsule.

Post-irradiation examination of the unclad carbide capsule started on June 3, 1963 in General Atomic Hot Cell. When the cask was opened,

the alarm in the chimney of the Hot Cell went off, indicating leakage of volatile fission products from the capsule, presumably due to the existence of an open path between the inside of one or more of the pins and the ambient atmosphere. As no such leak was detected during the irradiation, it was suspected that the open path developed when the capsule was sawed off from the lead tube. As no fission gases were detected in the annular space outside the pins and no rupture was detected in the Inconel wall of all three pins, it was concluded that the leakage path more likely passed from the inside of one or more of the pins through the W thermocouple well and the Ta sheath of the thermocouple to the points where the thermocouple sheath was sawed open. The exact reason for the development of such a leak, however, could not be pinned down until metallographic examinations of the contents of the pins were made. After the three pins were separated from the graphite thermal bond, they were individually put into a enclosed chamber inside the Hot Cell and the chamber was evacuated. More fission gases were detected in the exhaust when Pin No. 2 (high density pin) and Pin No. 3 (low density pin) were evacuated, but not when Pin No. 1 (medium density pin) was evacuated. Thus, it seemed that both Pin No. 2 and Pin No. 3 were definitely leaking, while Pin No. 3 might be leak-tight or might leak so badly that all the released fission gases were lost before the evacuation. A decision was reached to examine the high density pin (No. 2) first, followed by the low density pin (No. 3) and then the medium density pin (No. 1).

The following are the subjects involved in the post-irradiation examinations of the capsules:

(i) The macroscopic appearances of each sample.

(ii) The dimensions of each sample.

(iii) The microstructures of each sample, W thermocouple well, Ta thermocouple sheath and thermocouple.

(iv) Radiochemical analysis of selected samples for the burn up and the fission product retention.

(v) Radiochemical analysis of the materials condensed on the wall of the inner Inconel can.

(vi) Radiochemical analysis of sections of the dosimeter wires to determine the integral thermal and fast neutron fluxes at various locations along the cylindrical axis of the capsule.

By the end of this reporting period, the samples in Pin No. 2 and Pin No. 3 have been examined macroscopically and their dimensions determined. Studies of the microstructures of these samples are in progress. The results are summarized as follows:

Pin No. 2 (high density pin)

(i) No catastrophic cracking or fragmentation occurred in the irradiated samples.

(ii) Most of the samples cannot be removed from the W thermocouple well and the W spindle due to the sticking of some of the samples to the W thermocouple well and the presence of burrs on the W spindle.

(iii) Indentations formed by the W wire spacers were observed on both the cylindrical surface and the top and bottom surfaces of all the fuel samples, indicating the softness of these samples at high temperatures.

(iv) Sifnificant swelling occurred in all the fuel samples. In Table II the percentage increases of the outside diameters of all the samples in the high density pin upon irradiation are summarized.

Pin No. 3 (low density pin)

(i) One 90 UC - 10 ZrC depleted control sample (sample 4) and one 90 UC - 10 ZrC fuel sample (sample EL) became crumbled upon handling. It is believed that this was caused mainly by the leakage of the pin and the storage of the sawed-off capsule under water for a few days at the General Electric Test Reactor before loading into the cask. During this storage period, water vapor might have come into contact with these samples. Since 90 UC - 10 ZrC is more reactive than the samples of other compositions in the pin, especially in low-density porous form, reaction with water vapor might have considerably weakened these samples mechanically.

(ii) The 50 UC - 50 ZrC sample V-9 developed cracks. However, these cracks might have been hidden in the sample before irradiation.

(iii) All the other samples were intact and did not stick to the W thermocouple well or the W spindle.

(iv) Indentations left by W wire spacers were observed on both the cylindrical surface and the top and bottom samples of all the fuel samples, indicating the softness of these samples at high temperatures.

(v) Some swelling was observed in all the fuel samples. The results are summarized in Table III.

3.2 Clad Capsule.

Irradiation of the clad capsule started in the General Electric Test Reactor on June 23, 1963. All four high temperature thermocouples are functioning. Fig. 1 shows the thermocouple readings of the four pins in the capsule up to June 30, 1963. The reason for the sudden drop in the reading of the thermocouple in the clad high-density carbide pin is not clear at this moment. The irradiation will be completed by July 14, 1963 and hot cell examinations will be started during the first week of August, 1963.

4. Studies of New Cathode Materials.

4.1 Vacuum Emission and Vaporization.

The vacuum emission of sample D_1-3 (30 UC - 70 NbC, cold-pressed and sintered, ~74.5% dense on the basis of the theoretical density of the nominal composition) has been measured in the temperature range of 1650 - 1950°K after the determination of its vacuum rates of vaporization. The results are shown in Fig. 2. Fig. 3 summarizes all the vacuum emission data obtained under the present contract on carbide samples which have been thoroughly stabilized by thermal treatment during the vacuum vaporization runs. Some of the vacuum emission data described in the final report for Contracts NAS 5-1253[1] are included for comparison. The emission of the 30 UC - 70 NbC sample seems to be slightly higher than that of the other samples (except B_1-2). The data will be checked by using other 30 UC - 70 NbC samples. Studies of the vacuum rates of vaporization of a 30 UC - 70 TaC sample (D_1-4, cold-pressed and sintered) are underway.

4.2 Diffusion.

A diffusion run(D_2-5) of 24 hours duration has been carried out between hypostoichiometric 30 UC - 70 ZrC and Ir, Re and W-26Re at 1800°C.

The samples are being examined metallographically.

4.3 Diffusion-Emission.

Two vapor-deposited W clad samples, one (D_3-2) over hypostoichiometric UC (4.70 wt-% C) and the other (D_3-3) over stoichiometric 30 UC - 70 ZrC are being studied at 1800°C. The thickness of the W clad was about 30 mils in both cases. Up to the end of this reporting period, they have been at 1800°C for 371 and 356 hours, respectively. The results are summarized as follows:

Sample D_3-2.

Since it usually takes about 50 hours to outgas the sample and to bring the system into operating conditions, the first measurement on the vacuum emission was carried out after the sample had been at 1800°C for 59 hours. The Schottky plot corresponding to this measurement is shown in Fig. 4. It can be seen that the curve has a very steep "straight line" portion and that the emission at higher field strength decreases rather than increases with the increase of the applied voltage. The plot is thus not of the "normal" type, indicating the surface could have already been contaminated by fuel components diffusing through the clad. The shape of the plot changes gradually with the time of heating. The Schottky plot after 371 hours at 1800°C is also shown in Fig. 4. The straight line portion of the plot is broken into a number of steps and the emission increases continuously with the increase of the applied voltage. The emitting surface thus seems to be quite heterogeneous and does not represent that of pure W. The test is being continued for a period of 1000 hours.

Sample D_3-3.

Th Schottky plots of sample D_3-3 for various periods of time at 1800°C are shown in Fig. 5. Each of these curves exhibits two plateau regions. While the zero field emission for the first plateau region did not seem to change significantly with time of heating, the zero field emission for the second plateau increased with the time of heating. The former lies around 2 m.a./cm^2, while the latter increases from a little over 2 m.a./cm^2 after 56 hours at 1800°C to about 6 m.a./cm^2 after 350 hours at 1800°C, as compared to a value of 4 m.a./cm^2 for the vacuum emission value of W at 1800°C, as reported in the literature[4]. The test is being continued for a period of 1000 hours.

II. FUTURE PLANS.

1. Long-ter Tests.

1.1 Rate of Vaporization of UC-ZrC in Cesium Vapor.

The two cells containing the 30 UC - 70 ZrC and the 90 UC - 10 ZrC samples will be tested for periods of 1000 hours.

1.2 Emission Stability of UC-ZrC and W-Clad UC and UC-ZrC in Cesium Vapor.

The conditions for obtaining 5 watts/cm^2 will be established with the control cell. Efforts will be made to assemble the W-clad 30 UC - 70 ZrC and the bare 30 UC - 70 ZrC cells and to start the operation of these cells.

2. Studies of High-Temperature Properties of UC-ZrC.

2.1 Effect of Porosity on the Rate of Vaporization and Electron Emission of UC-ZrC in Vacuum.

Measurements will be started on the following samples:

(i) high density (~95% dense) 30 UC - 70 ZrC, hot pressed.

(ii) low density (~85% dense) 30 UC - 70 ZrC, cold-pressed and sintered.

(iii) low density (~85% dense) 90 UC - 10 ZrC cold-pressed and sintered.

The results, combined with those already available, should indicate how porosity would affect the vacuum emission and vaporization of UC-ZrC of these compositions.

2.2 Redeposition Studies.

It is expected that the physical redeposition studies of UC versus Ni anode will be concluded and chemical redeposition studies using CsF will be initiated.

2.3 Thermionic Emission Microscopy.

Studies of the activation, poisoning and regeneration of a 90 UC - 10 ZrC sample will be made.

2.4 High-Temperature Mechanical Properties.

Tests of the 10 UC - 90 ZrC samples will be made.

3. Irradiation Studies.

3.1 Unclad Carbide Capsule.

Hot-cell examinations of the contents of the unclad carbide capsule will be continued.

3.2 Clad Capsule.

The irradiation of the clad capsule in the General Electric Test Reactor will be completed and the capsule returned for hot cell examinations.

4. Studies of New Cathode Materials.

4.1 Vacuum Vaporization and Emission.

Measurements on the vacuum vaporization rates and the vacuum emission of 30 UC - 70 TaC (sample D_1-4) will be completed.

4.2 Diffusion.

The samples in diffusion run D_2-5 will be examined metallographically. Studies of ZrC as a diffusion barrier for the diffusion between UC and refractory metals will be initiated.

4.3 Diffusion-Emission.

Tests on sample D_3-2 and D_3-3 will be continued for periods of 1000 hours.

5. Fabrication Development.

No further work is planned on fabrication development during the rest of this contractual period.

III. REFERENCES.

1. Final Report for Contract NAS 5-1253, GA-3523.

2. Final Report for Contract NAS 3-2310, GA-3642.

3. Third Quarterly Report for Contract NAS 3-2532, GA-4391.

4. Haas, G. A., Thermionic Electron Sources, NRL Report 5657, U.S. Naval Research Laboratory, Washington, D. C.

5. Haas, G. A., and J. T. Jensen, Jr., Jrnl. Appl. Phys., 31, 1231 (1960).

Table I

Summary of Data Obtained from Physical Redeposition Studies

Sample No.	Run Number	Sample Temp(°K)	Ni Anode Temp(°K)	Rate of Vapor. in Absence of Anode / Rate of Vapor. in Presence of Anode	Total Amount Vaporized W(mgs)	Condensate Collected* (mgs)		
						W_U	W_{Zr}	$\frac{W_U+W_{Zr}}{W} \times 100$
B_2-1	1	2013	558	~1	37.20	35.00	-	94%
(UC)	2	2083	983	2.0	70.00	53.00	-	76%
	5	1948	683	1.5	6.99	6.14	-	88%
	6	2008	983	2.3	13.83	10.50	-	76%
B_1-10	3	2038	983	2.2	4.59	2.93	0.02	64%
(90UC-10ZrC)	4	2027	983	2.3	3.57	2.60	0.02	73%
B_1-9a	4	2123	983	~1	5.88	4.80	0.81	95%
(30UC-70ZrC)	5	2082	983	~1	4.20	3.38	0.60	95%

* The condensate on the anode surface was dissolved with dilute HNO_3 and the solution was analyzed for U and Zr content. However, the carbon content of the condensate cannot be determined due to the formation of volatile carbon compounds during the dissolution.

Table II

Change in the Outside Diameter of
the Samples in the High Density Pin (Pin No. 2)
Upon Irradiation

Specimen Number	Type	Density of Sample Before Irradiation (% of theoretical density of nominal comp.)	Percentage Change in the Original Outside Diameter Upon Irradiation*
10	ZrC	78	0
14	ZrC	78	0
B	90UC - 10ZrC (depleted control)	89.5	0
8	20UC - 80ZrC (enriched thermal shield)	94	+3.9
13	20UC - 80ZrC	97.6	+6.0
3X	20UC - 80ZrC	92.8	+6.5
AZ	50UC - 50ZrC	90	+7.4
16	20UC - 80ZrC	94.5	+7.5
AY	50UC - 50ZrC	95.2	+7.7
15	20UC - 80ZrC	93.2	+7.1
5	20UC - 80ZrC (enriched thermal shield)	93	+6.5
G	90UC - 10ZrC (depleted control)	90	0
11	ZrC	78	0
12	ZrC	78	0

*Since the cross sections of the irradiated samples are no longer circular due to deformation by swelling, the maximum dimension of the cross section was used for the calculation.

Table III

Change in the Outside Diameter of
the Samples in the Low Density Pin (Pin No. 3)
Upon Irradiation

Specimen Number	Type	Density of Sample Before Irradiation (% of theoretical density of nominal comp.)	Percentage Change in the Original Outside Diameter Upon Irradiation*
1	ZrC	78	0
2	ZrC	78	0
4	90UC - 10ZrC (depleted control)	88	crumbled upon handling
1	20UC - 80ZrC (enriched thermal shield)	95	+3.6
EB	90UC - 10ZrC	84	+1.6
V-1	50UC - 50ZrC	87.5	+4.8
18	20UC - 80ZrC	83.2	+3.8**
EL	90UC - 10ZrC	85	crumbled upon handling
V-9	50UC - 50ZrC	87	cracked
17	20UC - 80ZrC	83.6	+5.2**
6	20UC - 80ZrC (enriched thermal shield)	95	+3.0
L	90UC - 10ZrC (depleted control)	91	0
3	ZrC	78	0
13	ZrC	78	0

* The maximum dimension of the cross section of the irradiated sample was used for the calculation.

** Sample became hourglass shaped. It is suspected that for sample 18 the increase in the dimension of the surface facing sample V-1 was mainly due to the swelling of sample V-1, since the dimensions of the adjacent surfaces of these two samples were about equal. The swelling of sample V 1 was transmitted to the adjacent surface of sample 18 by the W spacer wire embedded in both surfaces due to their softness at high temperatures. The same relationship holds for sample 17 and the high density 20UC - 80ZrC thermal shield sample 6. In the latter case, the increase in the dimension of the surface of sample 17 opposite to sample 6 is believed to be due to the swelling of the high density sample 6. Metallographic examinations are underway to check these hypotheses.

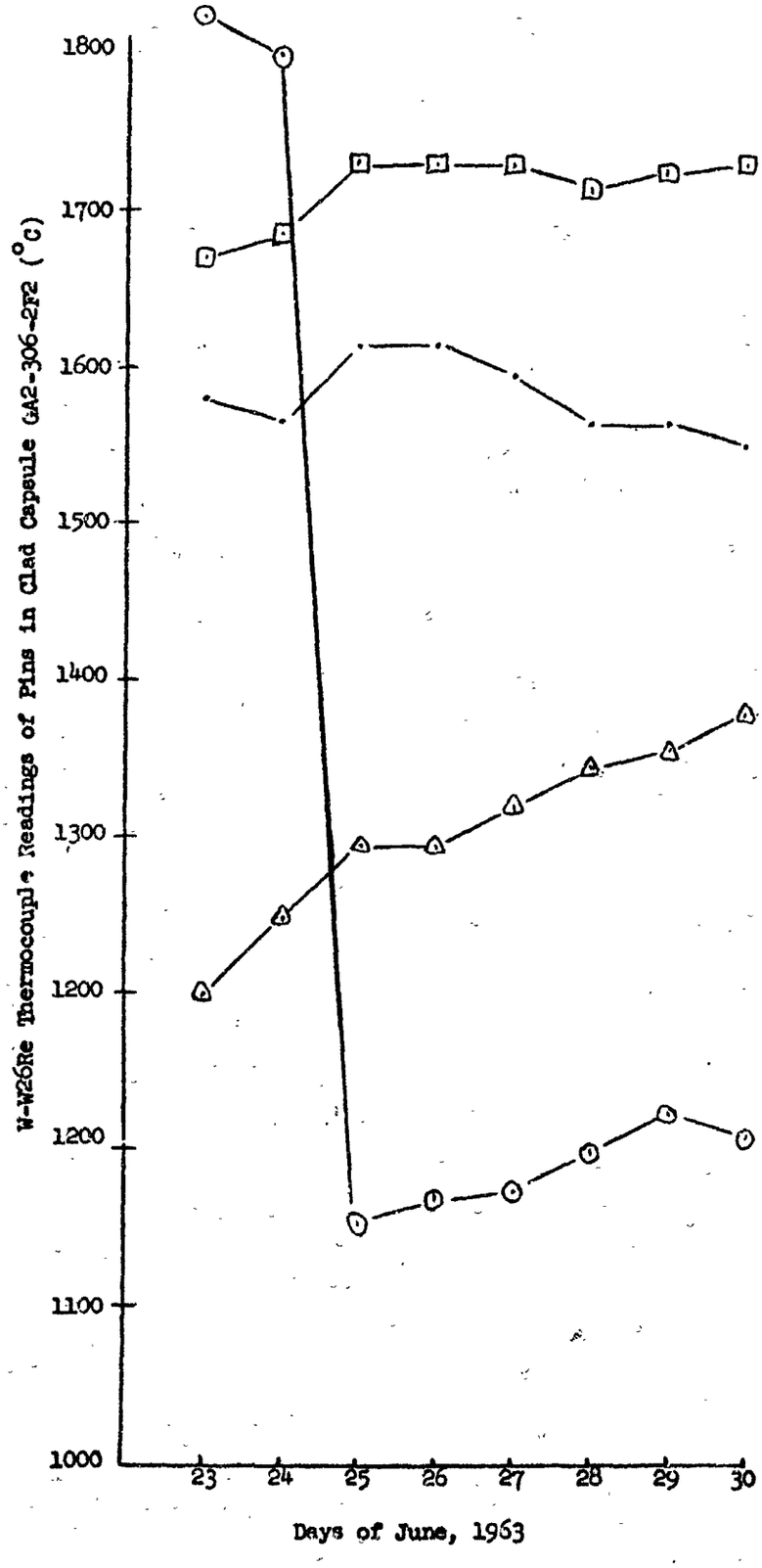

Fig. 1--Thermocouple readings of the four pins in the capsule up to June 30, 1963.

- W-W26Re T.C. No. 7 , Pin No. 1 Bottom cermet clad pin.
- W-W26Re T.C. No. 8 , Pin No. 2 High density carbide clad pin.
- W-W26Re T.C. No. 9 , Pin No. 3 Low density carbide clad pin.
- W-W26Re T.C. No. 10 , Pin No. 4 Top cermet clad pin.

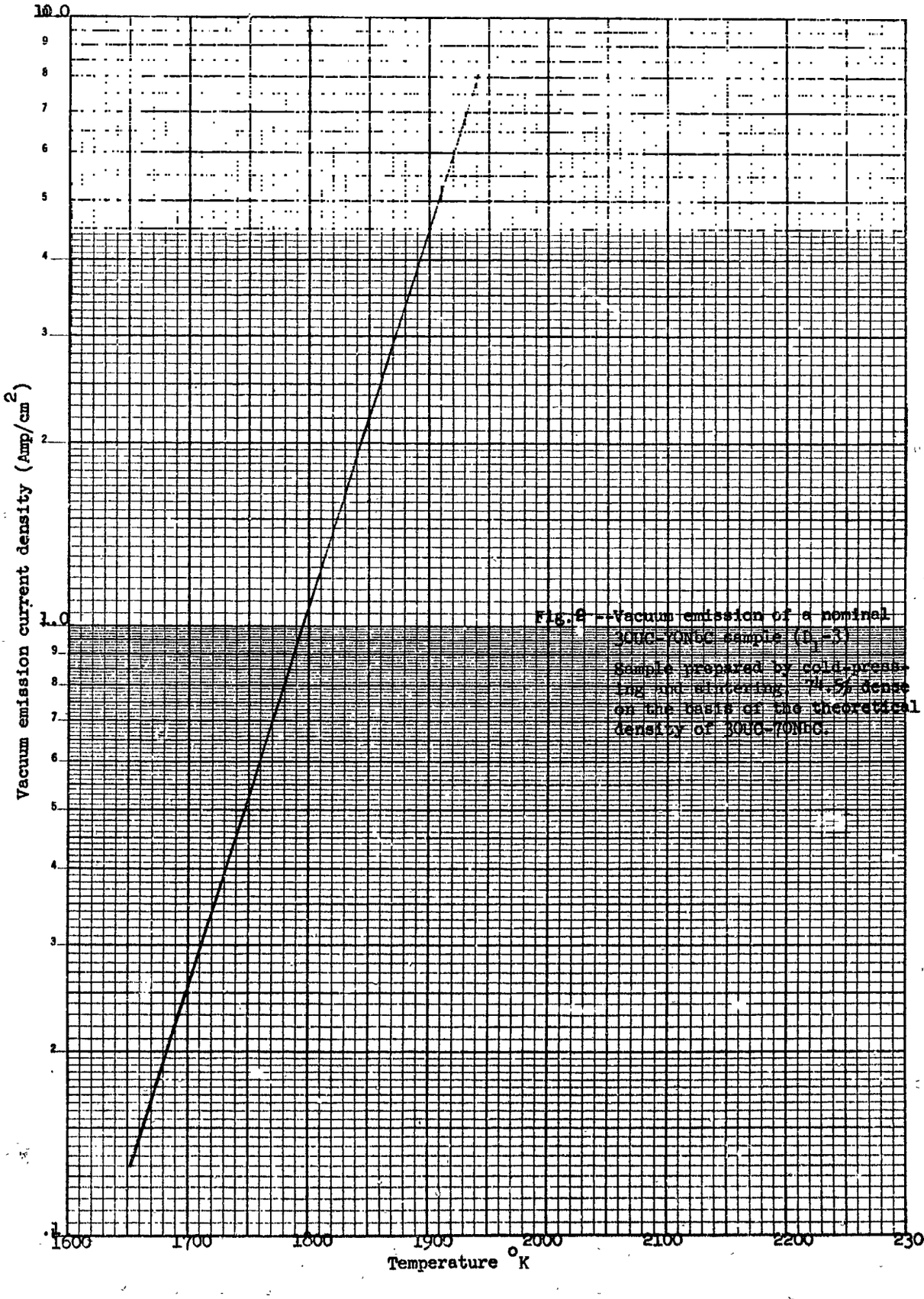

Fig. 6--Vacuum emission of a nominal 30UC-70NbC sample (D_1-3)

Sample prepared by cold-pressing and sintering; 74.5% dense on the basis of the theoretical density of 30UC-70NbC.

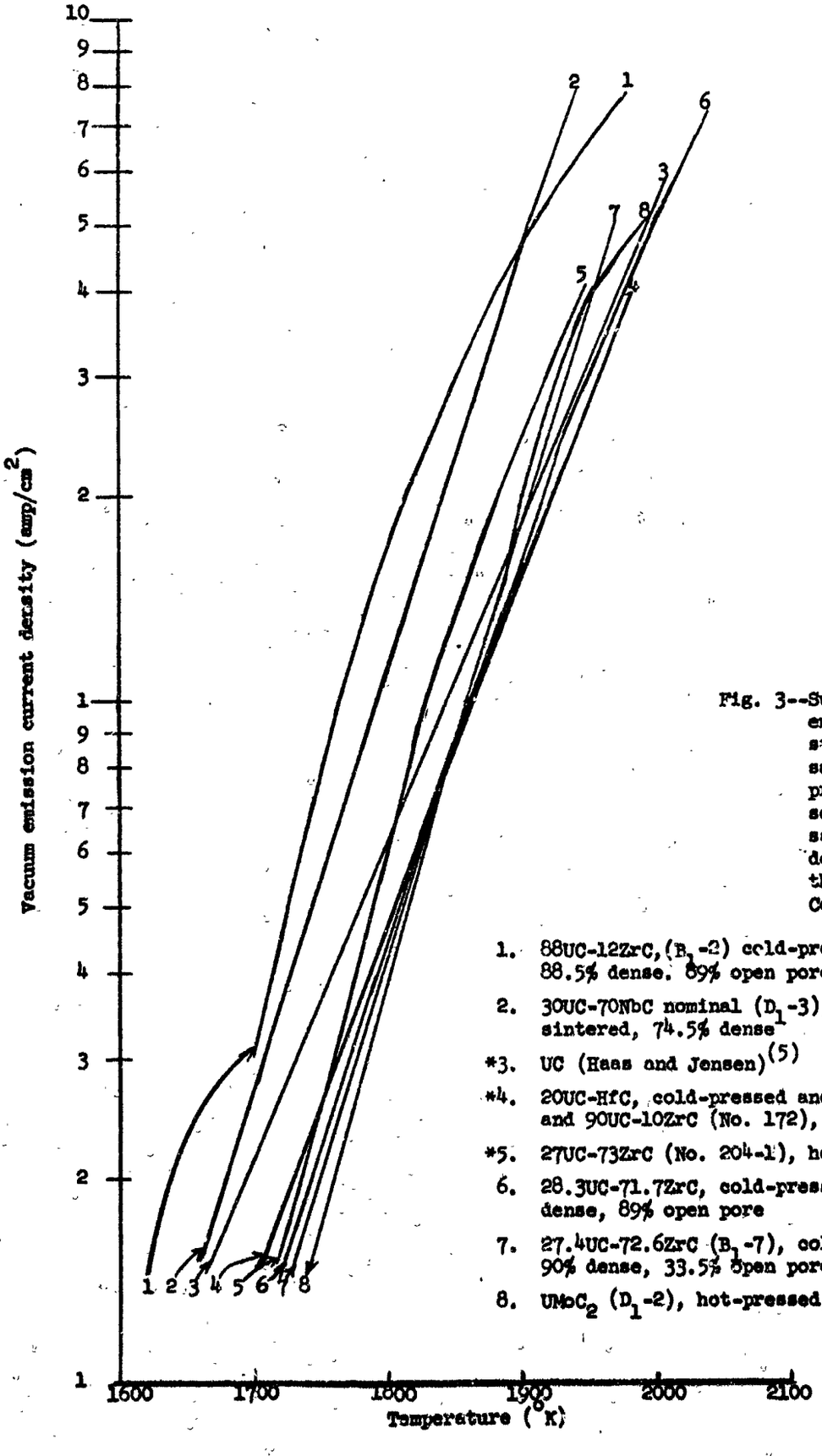

Fig. 3--Summary of the vacuum emission of thermally stabilized carbide samples studied in the present contract and some other carbide samples (marked with*) described previously in the work under Contract NAS 5-1253.

1. 88UC-12ZrC, (B_1-2) cold-pressed and sintered, 88.5% dense. 89% open pore
2. 30UC-70NbC nominal (D_1-3), cold-pressed and sintered, 74.5% dense
*3. UC (Haas and Jensen)[5]
*4. 20UC-HfC, cold-pressed and sintered, ~ 70% dense and 90UC-10ZrC (No. 172), cast, ~95 % dense
*5. 27UC-73ZrC (No. 204-1), hot-pressed, 95.5% dense
6. 28.3UC-71.7ZrC, cold-pressed and sintered, 86.7% dense, 89% open pore
7. 27.4UC-72.6ZrC (B_1-7), cold-pressed ans sintered, 90% dense, 33.5% open pore
8. $UMoC_2$ (D_1-2), hot-pressed, 88.9% dense.

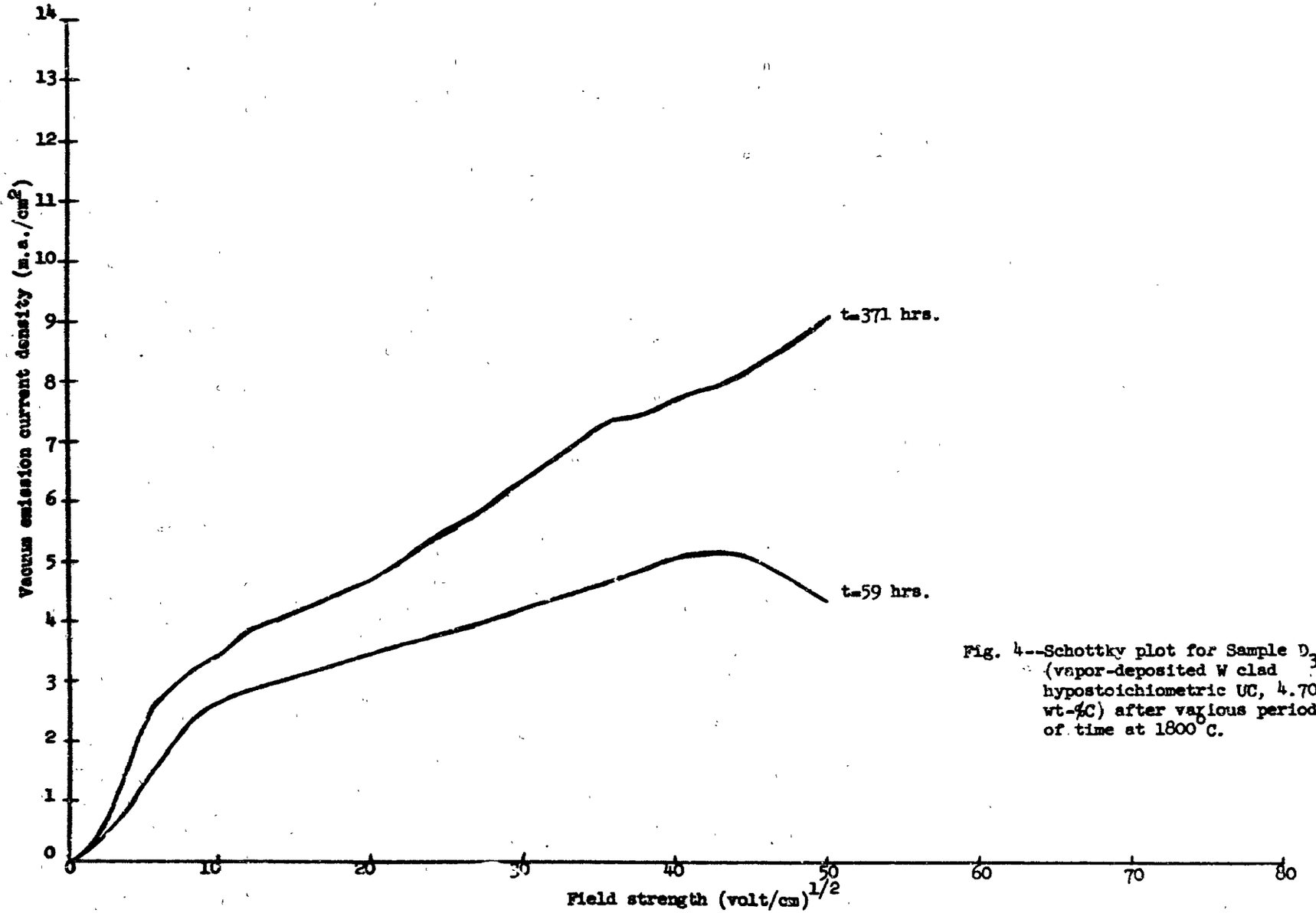

Fig. 4--Schottky plot for Sample D_3-2 (vapor-deposited W clad hypostoichiometric UC, 4.70 wt-%C) after various periods of time at 1800°C.

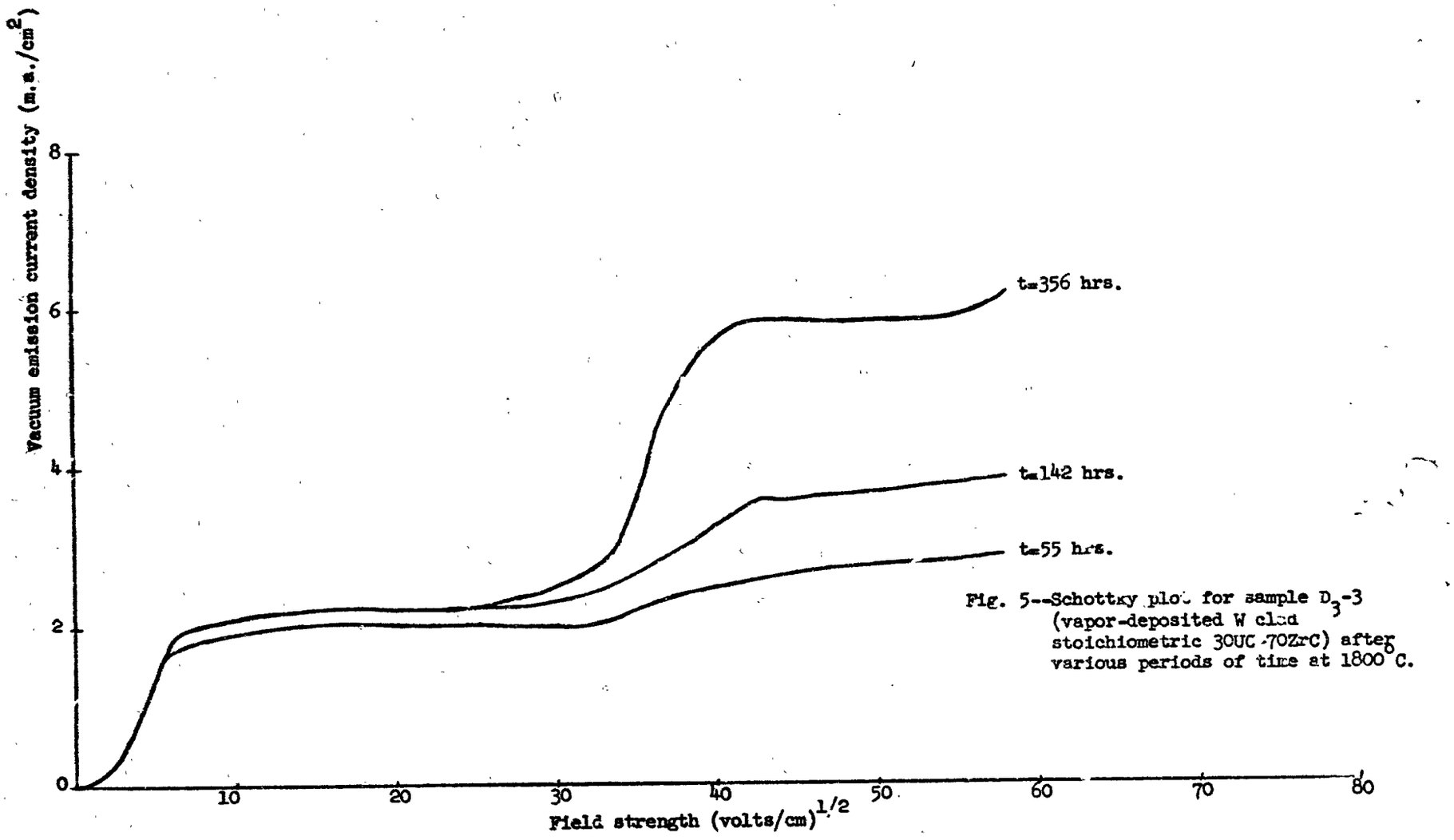

Fig. 5--Schottky plot for sample D_3-3 (vapor-deposited W clad stoichiometric 30UC-70ZrC) after various periods of time at 1800°C.